SOP #031601: TOTAL DISSOLVED SOLIDS
(Also called: Total Filterable Residue)
METHOD #: 2540C

Revision: 5
Date: 05/25/10

Location: QA Officer's Office
SOP Files
TCLP Laboratory
Solids Laboratory

1.0 Scope

1.1 To describe the method for measuring Total Dissolved Solids (TDS).

2.0 Summary of Method

2.1 An aliquot of sample is taken from a stirring sample and filtered through a filter (pre-weighed filter if TSS is also needed) into a clean side arm flask. After rinsing the solids retained on the filter, the filtrate is quantitatively transferred to a weighed evaporating dish. The sample is then evaporated at 103°C in the oven. Then the dish + solids is moved to a 180°C oven (usually a muffle oven) and dried to a constant weight (usually at least one hour). The weight of solids in the dish represents the TDS.

3.0 Interferences

3.1 Poor shaking or stirring can lead to wrong results.
3.2 Improper drying of the solids (not to constant weight) will give higher results than the true value.

4.0 Apparatus and Materials

4.1 If only TDS is needed use un-weighed glass fiber filters. If TSS is also needed, use Proweigh filters Catalogue number: F934447MM from Environmental Express (phone: 800-343-5319) or www.envexp.com or. Note: if TVSS are to be analyzed TVSS pre-primed (yellow) filters must be used (catalogue number: F93447VOL from Environmental Express). Or the TSS filter can be primed at 550 °C.
4.2 Vacuum filtering apparatus compatible with 47 mm glass fiber filters.
4.3 25 mL large mouth Pipets
4.4 Graduated cylinders: 50 mL, 100 mL.
4.5 150 mL aluminum or ceramic evaporating dishes
4.6 Oven
4.7 Muffle Furnace
4.8 Thermometer
4.9 Dessicator
4.10 Analytical Balance
4.11 Vacuum pump
4.12 Tweezers

5.0 Reagents

5.1 DI Water
5.2 LCS from ERA.

6.0 Sample Collection, Preservation and Handling

6.1 The sample is usually a composite sample collected in a plastic or glass liter bottle with no preservation other than refrigeration at 4±2°C. The holding time is 7 days from sample collection.

7.0 Procedure

7.1 Record the date, and time of analysis as well as your initials as the analyst on the bench sheet and in the appropriate batch for TDS. A batch is defined as 10 samples of the same or similar matrix.
7.2 If TSS is also needed, record the tin number and the filter weight of a preweighed 47 mm glass fiber filter on the TSS bench sheet and record the weight of the evaporating dish on the

Merit TDS bench sheet on the line indicating "Blank". If only TDS is needed record only the evaporating dish weight on the TDS bench sheet. Using tweezers place the filter in the filter holder with the wrinkled side up.

7.3 Place the filter holder assembly in the clean 150 mL side arm flask with the vacuum hose attached to the side arm.

7.4 Turn on the vacuum and filter 100 ml of deionized water through the filter until all of the water is drawn through. This is the Blank.

7.5 Record the 100 ml volume on the bench sheet(s) for the Blank.

7.6 Then remove the filter, if TSS is needed, and place it in its tin. Then place tin and filter in a drying oven at 103^{o}C for one hour or more. Note: If Filters are dried for more than four hours you can be confident they are dried to constant weight.

7.7 Transfer the filtrate to its weighed evaporating dish and dry in 103^{o}C oven overnight.

7.8 Then move the ceramic evaporating dish to a 180^{o}C oven for at least 1 hour until dried to a constant weight.

7.9 Remove the ceramic evaporating dish from the oven and place it in a desiccator to cool to room temperature.

7.10 Weigh it to the nearest 0.1 mg using an analytical balance.

7.11 Record the weight on the bench sheet for the blank.

7.12 Record on the bench sheet the tin number and the filter weight for another filter and place the prewieghed filter in the filter holder assembly with the wrinkled side up, apply vacuum and wet the filter with a little deionized water to ensure adhesion to the holder.

7.13 Record the Merit number on the bench sheet. Then mix a sample by shaking it violently and quickly pour about 150 ml to 250 ml into a plastic cup or beaker.

7.14 Place a Teflon covered magnetic stirring bar in the cup and place the cup on top of a magnetic stirrer. Stir for one minute or more.

7.15 Using a 25 ml serological pipette with a wide opening, remove 25.0 ml of well mixed sample from half way into the cup and halfway between the mixing vortex and the side of the cup. Then drain the pipette onto and through the filter.

7.16 Repeat pipetting (step 16) until 100 ml has been filtered. 50 ml can be used if you know there are a lot of dissolved solids in the sample (from historical testing information).

7.17 Note that shaking vigorously and quickly pouring the sample from the bottle into a graduated cylinder will be a better sampling procedure for some samples. When using this method, pour the sample on to the filter from the cylinder.

7.18 Record the volume you filtered on the bench sheet.

7.19 As you did with the Blank, remove the filter from the filter holder, place it back in its tin for TSS and dry it in a 103^{o}C oven for one hour or more.

7.20 Transfer the filtrate to its weighed evaporating dish and dry it at 103^{o}C overnight. Then dry the dish in a 180^{o}C oven to a constant weight.

7.21 Remove the tin from the oven and cool it to room temperature in a Desiccator. Carefully weigh it on an analytical balance to 0.1 mg.

7.22 Record the weight on the bench sheet.

7.23 Check the dryness of the sample by drying it for another 15 minutes. Then reweigh. The sample is dried to a constant weight if the two weights differ by no more than 4 % or 0.5 mg which ever is less.

7.24 Record the weight on the bench sheet.

7.25 Be sure to perform the QC analyses summarized in Section 8.0.

7.26 Repeat Steps 13 through 24 for each sample.

8.0 Calculations

8.1 TDS mg/l = [[(Grams of dish + residue) – (Grams dish)]/ml sample filtered] X 1000 ml/L X 1000 mg/Gram

9.0 Quality Control

9.1 Also perform the following Quality Control for each sample set. A sample set is a group of one to ten samples run at one time.

Quality Control

QC Sample	Frequency	Acceptable Limits	Corrective Action	Follow up Corrective Action
Blank	1/10 samples	-3 to +3 mg/l	Redry and Reweigh	Reset the entire set of samples with new filters
Laboratory Control Sample-ERA Small Lab Minerals	1/10 samples	Within Supplier's Limits	Redry and Reweigh	Reset the entire set of samples with new filters
Sample Duplicate	1/10 Samples	+/- 20 % of average	Reset sample duplicates	Footnote non-homogeneity
Analytical Balance Check with "S" Weights	1/day	For 1 gram Weight +/_ 0.0008 g	Recalibrate Balance and Reweigh	Call for Service
Constant Weight Verification	1/10 samples	4 % or 0.5 mg which ever is smaller	Dry longer and reweigh.	Dry longer and reweigh.

9.0 Documentation

9.1 Total Dissolved Solids Bench sheet
- 9.1.1 Date Started
- 9.1.2 Date Finished
- 9.1.3 Time
- 9.1.4 Analyst
- 9.1.5 Date Checked
- 9.1.6 Checked by
- 9.1.7 Batch #
- 9.1.8 Merit #
- 9.1.9 TSS (Yes/No)
- 9.1.10 Tin #
- 9.1.11 mLs of Sample
- 9.1.12 g Tin
- 9.1.13 g Dry Solids + Tin
- 9.1.14 g Weight after 15 Minutes
- 9.1.15 Ratio: TDS/ Conductivity
- 9.1.16 TDS (mg/L)
- 9.1.17 % Recovery
- 9.1.18 % RPD

9.2 Oven Temperature Log

9.3 TDS/TSS Excel File

10.0 References

10.1 *Standard Methods*, twentieth edition, Method 2540C, Solids.

11.0 Safety

11.1 Eye protection and gloves must be worn while performing TDS analyses.

11.2 Every Laboratory area has eyewash, emergency shower, and fire extinguisher. The metals lab also has dust masks available for use with dust samples.

11.3 The air system through out the laboratory area is on a 100% fresh air exchange system, this system exchanges 100% the air in the laboratory area with air from outside 6 times per hour and 30 times per hour when the emergency purge button is hit.

11.4 A reference file of material safety data sheets (MSDSs) is available to all personnel.

12.0 Waste Disposal and Pollution Prevention

12.1 All laboratory waste must be managed, stored, and disposed in accordance with all federal and state laws and regulations.

12.2 Additional information can be found in the Sample Disposal SOP and Merit's Waste Management Plan and Handbook.

13.0 Approval and Issue

13.1 This section indicates which personnel have read, accepted, and approved the SOP. All analysts involved with the SOP should acknowledge their comprehension of the SOP with a signature and a date.

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Analyst Date

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Andy Ball, QA Officer Date

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Maya V. Murshak, Technical Director Date